Standard Operating Procedure for GLNPO Turbidity: Nephelometric Method

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1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters.

1.2 The working range is 0-20 NTU. Samples more turbid than 20 NTU can be determined by appropriate dilution.

2.0 Summary of Method

The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. The design of the nephelometer is specified in the method. A standard suspension of Formazin is used for calibration.

3.0 Sample Handling and Preservation

Samples are analyzed immediately or stored at 4°C. They are considered stable for at least 48 hrs when stored at 4°C.

4.0 Interferences

4.1 The presence of floating debris and coarse sediments will give high readings.

4.2 Air bubbles will cause high results.

4.3 Colored samples will cause low results.

5.0 Apparatus

5.1 The turbidimeter shall consist of a nephelometer with light source for illuminating the sample and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be so designed that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.

5.2 The sensitivity of the instrument should permit detection of a turbidity difference of 0.02 unit or less in waters having turbidities less than 1 unit. The instrument should measure from 0 to 20 units turbidity. Several ranges may be necessary to obtain both adequate coverage and sufficient sensitivity for low turbidities.

5.3 The sample tubes to be used with the available instrument must be clear, colorless glass. They should be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. They must not be handled at all where the light strikes them, but should be provided with sufficient extra length, or with a protective case, so that they may be handled. Differences in physical design of turbidimeters will cause differences in measured values for turbidity even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed.

5.4 Light source: Tungsten lamp operated at a color temperature between 2200-3000°K.

5.4.1 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.

5.4.2 Detector: Centered at 90° to the incident light path and not to exceed ±30° from 90°. The Detector, and filter system if used, shall have a spectral peak response between 400 and 600 nm.

5.5 The Hach Turbidimeter Model 2100 and 2100A, is in wide use and has been found to be reliable, however, other instruments meeting the above design criteria are acceptable.

6.0 Reagents

6.1 Reagent water: All reagents are prepared using water which has passed through at least two ion exchange cartridges. Throughout this SOP, water is understood to mean reagent water unless otherwise specified, and dilute, used as a verb, means dilute with reagent water.

6.2 Stock formazin turbidity suspension:

Solution 1: Dissolve 1.00 g hydrazine sulfate, $(NH_2)_2 \cdot H_2SO_4$, in water and dilute to 100 mL in a volumetric flask.

Solution 2: Dissolve 10.00 g hexamethylenetetramine in water and dilute to 100 mL in a volumetric flask.

In a clean dry 100 mL volumetric flask, mix 5.0 mL (volumetric pipet) of solution 1 with 5.0 mL (volumetric pipet) of Solution 2. Allow to stand 24 hours at 25 ±3°C, then dilute to 100 mL and mix. Prepare monthly.

6.3 Standard formazin turbidity suspension:

Working standards can be prepared by dilution of the following quantities of the stock formazin turbidity suspension (nominal 400 NTU) to 200 mL.

Dilute to 200 mL	Resultant NTU
10 mL	20
5 mL	10
2 mL	4
0.5 mL	1
0.2 mL	0.4
0.0 mL	0

7.0 Procedure for Turner Designs Model 100 Nephelometer

7.1 The instrument must be switched on and allowed to warm up for at least one half hour prior to use.

7.2 Monthly the 1 X range and the 10 X range should be correlated by initially adjusting the calibrate knob so that a 10.0 standard reads 9.80 on the 1 X range. The range is then switched to 10 X and the top screw inside the front door is adjusted until the reading is 9.8. Check the 1 X range to verify that the reading is still 9.80.

7.3 Monthly and as necessary to preclude zero readings for positive turbidity samples(the meter will not display negative readings), zero turbidity must be set to assure positive readings. With the cell removed from the holder and the range set to 1 X, adjust the lower screw inside the front door so that the digital readout is between 0.01 and 0.05 units.

7.4 Initially and for each lake or weekly (whichever comes first) a geometric series of calibration standards prepared as above must be used to define a calibration curve. A sealed reference 20 NTU commercial standard is used to obtain a readout of 20.0 by adjusting the calibrate knob. Readings are then made on the freshly prepared formazin standards.

7.5 Prior to taking a series of readings, the reference 20 NTU commercial standard will be used to set the readout to 20.0. If the reference 20 NTU standard is lost, prepare or otherwise obtain a new 20.0 NTU reference and proceed to Step 7.4.

7.6 Except for the 20 NTU commercial reference standard all readings should be made using the same sample cell. The sample cell should contain reagent water when not in use. It should be handled in a manner to preclude touching it where the light strikes it. It should be discarded and the machine re-standardized when it becomes scratched or etched.

7.7 Readings for samples and calibration standards over 6.0 NTU should be made on the High Range and those under 6.0 NTU should be made on the Low Range.

7.8 An aliquot of the sample warmed to 25°C is used for the turbidity measurements to preclude condensation on a cold sample cell. The condensation would cause erroneous readings.

7.9 Readings may be taken immediately. If the turbidity declines continuously such as with waters from the Niagara plume it is assumed that the initial readings are as correct as any that will be obtained. If a reading is variable, however, such as is found with a piece of debris, a second aliquot can be used for the reading.

7.10 If the turbidity is over 20.0 it can be diluted 1:1, 1:3, 1:7, 1:15 etc. to obtain a reading and the appropriate factor applied to the intermediate result to determine the actual turbidity.

8.0 Calculations

8.1 Use linear regression on the results from the calibration standards to generate a calibration curve.

8.2 The results are not edited by the analyst. If the result is -0.02 then that result is reported. The computer program rounds the results to 0.01 NTU.

9.0 Quality Control

9.1 Turbidity

Two Control Standards are run once per 12 hour shift, or once every two stations, whichever is less. The check standards are 10 NTU and 0.5 NTU, presently obtained from Advanced Polymers Systems. A reagent blank (reagent water processed through the sample storage container) is run approximately once in every four stations.

10.0 Preventive Maintenance

10.1 The cuvet should only be handled at the top **a** and efforts should be made to preclude spilling the sample or standard on the outside of cuvet, which will necessitate drying the cuvet with a clean soft dry tissue.

10.2 A separate bottle of verified low turbidity reagent water should be maintained exclusively for laboratory blanks and working standards preparation. When it shows signs of deterioration it should be replaced.

11.0 Troubleshooting/Corrective Action

11.1 A dirty or scratched cuvet should not be used. If reagent water gives a reading 0.10 units more than the empty compartment, then the water is turbid or the cell needs to be cleaned or discarded. A blank reading that is 0.03 to 0.05 units more than the empty compartment is not unusual.

11.2 The source of excessive background readings can sometimes be identified by opening the

front door of the instrument and observing the cuvet in place.

12.0 References

12.1 EPA Publication, March 1979. "Methods for Chemical Analysis of Water and Wastes". EPA #600/4-79-02.

12.2 Standard Methods for the Analysis of Water and WasteWater, 16th Edition APHA-AWWA-WPCF.

12.3 Instruction manual for Turner Designs Model 100 Nephelometer.